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Published in:
Applied Physics Letters

Link to article, DOI:
[10.1063/1.1984092](https://doi.org/10.1063/1.1984092)

Publication date:
2005

Document Version
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):
Davis, Z. J., & Boisen, A. (2005). Aluminum nanocantilevers for high sensitivity mass sensors. *Applied Physics Letters*, 87(1), 013102. <https://doi.org/10.1063/1.1984092>

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Aluminum nanocantilevers for high sensitivity mass sensors

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(Received 8 February 2005; accepted 24 May 2005; published online 27 June 2005)

We have fabricated Al nanocantilevers using a simple, one mask contact UV lithography technique with lateral and vertical dimensions under 500 and 100 nm, respectively. These devices are demonstrated as highly sensitive mass sensors by measuring their dynamic properties. Furthermore, it is shown that Al has a potential higher sensitivity than Si based dynamic sensors. Initial testing of these devices has been conducted using a scanning electron microscope setup where the devices were tested under high vacuum conditions. The Q factor was measured to be approximately 200 and the mass sensitivity was measured to 2 ag/Hz by depositing electron-beam-induced carbon at the end of the nanocantilever. © 2005 American Institute of Physics. [DOI: 10.1063/1.1984092]

Cantilever based dynamic sensors have in recent years been a platform for highly sensitive mass sensors^{1–3} and have demonstrated higher sensitivities than commercial quartz microbalance technologies.⁴ A cantilever based mass sensor works by measuring changes in its resonant frequency due to mass changes. Intuitively, by decreasing the size of the cantilever it is possible to measure smaller mass changes. The mass sensitivity can be derived using the equation for the resonant frequency and is given by:^{1–3,5}

$$\left| \frac{\partial f}{\partial m} \right| = \frac{f}{2m} \Leftrightarrow \left| \frac{\Delta m}{m} \right| = 2 \frac{\Delta f}{f}, \quad (1)$$

where f is the resonant frequency and m is the mass of the resonator. This equation confirms that by decreasing the mass and increasing the frequency of the mechanical resonator the mass sensitivity is increased.

Current published works have reported ag/Hz mass sensitivity.^{1,3} This has been possible by miniaturizing the cantilever by means of using the emerging nanolithography techniques such as electron beam lithography on single crystal Si substrates.^{6,7} In this work we report on Al nanocantilevers as a suitable replacement for Si based mechanical resonators. It will be shown that higher mass sensitivities can be achieved using Al and a very simple process flow is presented, which uses standard contact UV lithography to achieve sub 500 nm wide, 100 nm thick cantilevers, which can fabricate thousands of nanocantilevers on a wafer scale. Finally, initial testing of the Al nanocantilevers using a scanning electron microscope setup will be presented, which has been used to measure Q factor and mass sensitivity.

By looking at the device as a whole, the cantilever must be excited into resonance in order to detect the change in resonant frequency. Making the cantilever very short would increase the mass resolution of a cantilever based sensor; however, this is not a viable design. By decreasing the length of the cantilever the stiffness is increased and thus larger forces are needed for dynamic actuation. However, by rewriting the equation for the first mode resonant frequency of a rectangular cantilever $f=0.162\sqrt{E/\rho \cdot t/l^2}$,⁷ it can be shown that the resonant frequency can be increased by decreasing

both the Young's modulus (E) and the density (ρ) of the cantilever material, if the thickness (t), width (w), and spring constant (k) of the cantilever are unchanged

$$f = 0.408 \frac{k^{2/3}}{t w^{2/3}} \cdot \frac{1}{\rho^{1/2} E^{1/6}}. \quad (2)$$

In Table I the mass sensitivity ($\partial m/\partial f$) is calculated for cantilevers of different materials. The spring constant of each cantilever is kept constant by adjusting only the length of the cantilever and maintaining a width of 1 μm and a thickness of 100 nm. By looking at Table I it is seen that Al has the best mass sensitivity followed by poly-Si and then single crystal Si. This is because Al has both a low density and low Young's modulus compared to the other cantilever materials. Among these materials, which are readily available for microelectromechanical systems (MEMS) fabrication, Al is the best choice.

In Fig. 1 the fabrication of the Al nanocantilevers is shown, which is based on a lift-off technique. The devices are realized on 4 in. Si wafers, Fig. 1(a). First, a resist mold is formed on the Si substrate using UV lithography, Fig. 1(b). Then, Al is deposited using electron (e)-beam evaporation, Fig. 1(c). Next, the Al deposited on the resist is removed by lift-off, Fig. 1(d). Finally, the metal structure is released by dry etching the underlying Si using an isotropic SF_6 based dry etch, Fig. 1(e).

The most critical step in the fabrication is the definition of the resist mold. In order to achieve sub 500 nm cantilever widths a reverse lithographic process with AZ5214 photoresist was used.⁸ Another important aspect of this process is

TABLE I. The calculated length, resonant frequency, and mass sensitivity for different cantilever materials. The cantilevers have a spring constant of 0.1 N/m, a thickness of 100 nm, and a width of 1 μm .

Material	E (GPa)	ρ (g/cm ³)	l (μm)	f (MHz)	$\partial m/\partial f$ (ag/Hz)
Al	74	2.70	5.70	2.61	1.18
Poly-Si	160	2.33	7.37	2.49	1.45
Si(100)	180	2.33	7.66	2.44	1.43
Ti	110	4.51	6.50	1.89	3.07
Cr	140	7.19	7.04	1.44	6.98
Ni	200	8.90	7.94	1.22	11.48
Au	80	19.30	5.85	0.96	23.19

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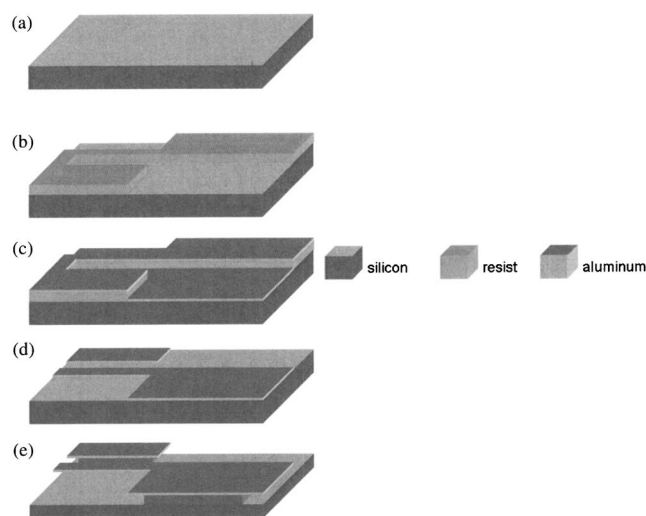


FIG. 1. Process flow of Al nanocantilevers.

that the release step is performed by dry etching, which alleviates stiction problems that are often seen using wet release techniques.

In Fig. 2 scanning electron images (SEM) are shown of the Al cantilever devices before and after dry underetching. From the images it is seen that the width of the nanocantilever is below 500 nm and the thickness is approximately 100 nm. The length of the realized nanocantilevers range from 1 to 10 μm .

In order to characterize the Al nanocantilevers in a vacuum environment the devices were wire bonded to a PCB substrate and placed inside a SEM. Electrical feedthroughs in the SEM facilitate electrical connection to the Al nanocantilever chips. On the chip an ac and dc voltage is applied between the driver electrode and the Al nanocantilever [Fig. 2(b)], which will actuate vertical motion. Vertical actuation is achieved because the electric field is nonuniform above and below the cantilever due to the Si substrate. Moreover, the released nanocantilever bends slightly upwards due to internal stress, which also creates a nonuniform electric field. Two SEM based methods of measuring the frequency response of the nanocantilever were used: (1) Sweeping the actuation voltage frequency and visually observing the vibrational amplitude of the nanocantilever and (2) focusing the e beam on the cantilever and monitoring the

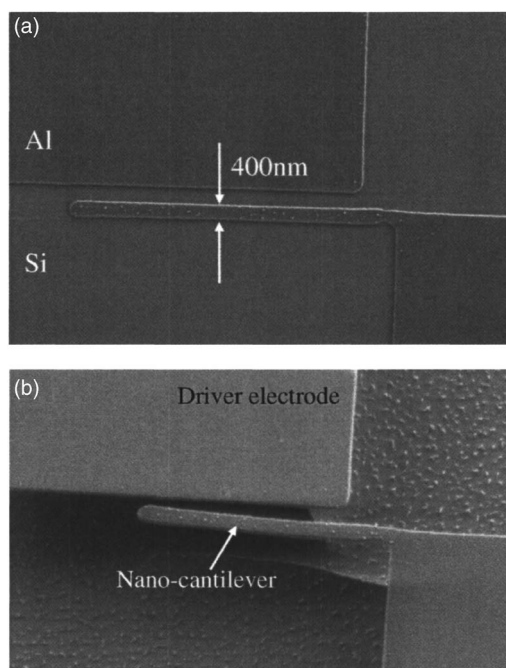


FIG. 2. SEM images of Al nanocantilever before (a) and after (b) dry underetching.

secondary electron detector signal as a function of the actuation voltage frequency.

In Fig. 3 two SEM images of an Al nanocantilever are seen at two different actuation frequencies. In this experiment an ac actuation voltage of 9 V pp was used. In Fig. 3(a) the nanocantilever is not in resonance and the image of the cantilever is sharp, whereas in fig. 3(b), at an actuation frequency of 1.43 MHz, the end of the cantilever is blurred due to the vibration. In Fig. 3(c) the frequency response of this device is seen by measuring the amplitude directly from the images taken at different actuation frequencies. From the SEM images the vibrational amplitude can be measured with an accuracy of approximately 30 nm. In order to measure the Q factor a Lorentzian fit was made and the Q factor was extracted. In this case the Q factor was measured to be approximately 200. Considering the high vacuum conditions this Q factor is very low. However, several factors can contribute to this, such as poor anchoring conditions due to underetching of the support [see Fig. 2(b)] and surface losses

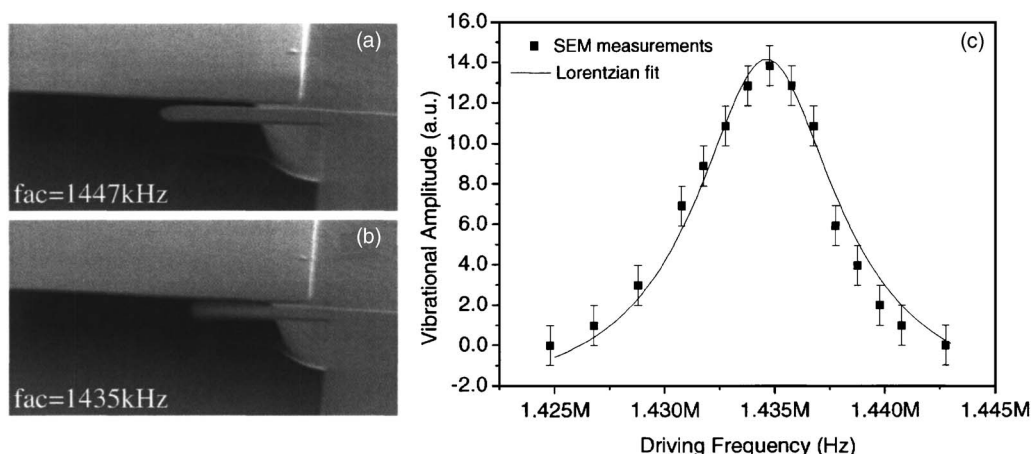


FIG. 3. SEM images of a nanocantilever out of resonance (a) and in resonance (b). (c) The measured frequency response and a Lorentzian fit. The cantilever is vibrating vertically.

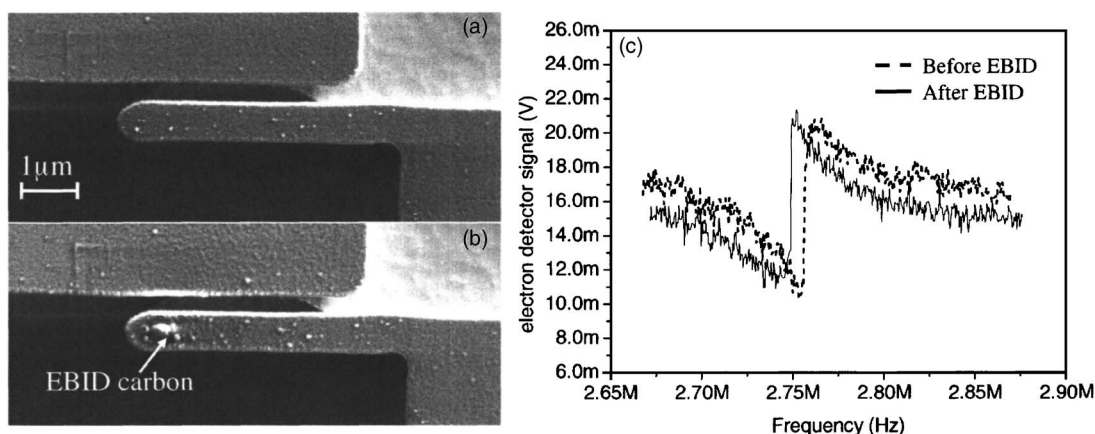


FIG. 4. SEM images of a nanocantilever before (a) and after (b) carbon deposition and (c) the measured frequency responses both before (dashed) and after (full) carbon deposition.

due to the large surface to volume ratio of these nanocantilevers.^{9,10}

By using the second measurement method the frequency response was measured using the exact same device as the previous experiment. In Fig. 4(c) the detector signal is seen to shift abruptly at an actuation frequency of 2.76 MHz. The reason for this abrupt shift is not fully understood, however, it could be due to a nonlinear behavior that is seen for large vibrational amplitudes.¹¹ Another aspect of this measurement is that the resonant frequency is measured here to be approximately double the first experiment. The reason is because in the first experiment the actuation of the cantilever was done with a pure ac signal between the driver electrode and cantilever, thus the actuation happened at half the mechanical frequency since the forces are proportional to the voltage squared. In the second experiment a dc voltage of 10 V and an ac voltage of 1 V were applied, thus the electrical and mechanical frequencies are the same. The theoretical expected resonant frequency (neglecting spring softening effects) of this Al cantilever is approximately 3.0 MHz, which corresponds well to the experimental values. The slight difference in the measured and theoretical values can be attributed to electrostatic spring softening and added mass during SEM imaging.

Finally, a mass measurement has been performed depositing carbon by electron-beam-induced deposition (EBID).¹² In Fig. 4 images of the Al nanocantilever are seen both before (a) and after (b) the EBID. The frequency responses of the nanocantilever before and after are shown in Fig. 4(c). By measuring the frequency shift (6.5 kHz) and using a rough estimate of the mass of the carbon deposit (14 fg) the mass sensitivity of the device is calculated to approximately 2 ag/Hz. The expected theoretical value, using Eq. (1), is approximately 1 ag/Hz, which is in good agreement with the experiment. In this case the minimal frequency shift (df) that can be measured is approximately 500 Hz, which leads to an absolute minimal detectable mass of approximately 1 fg in these experiments. However, this number can be improved by improving the Q factor and optimizing the electron detector signal with respect to noise.

It has been demonstrated that using a simple batch process, cantilevers with sub 500 nm widths can be fabricated on a wafer scale. Furthermore, Al nanocantilevers can be vertically excited into resonance with a lateral electrode. Through a simple characterization setup, involving a SEM, the Q factor was measured to approximately 200, which is lower than expected. However, more work needs to be done to investigate why, by improving current anchoring conditions, etc. Finally, measurements of EBID carbon have been done and a mass sensitivity of 2 ag/Hz has been measured. The SEM characterization technique has shown to be a very versatile and fast tool for measuring nanocantilevers in vacuum condition. Furthermore, this work demonstrates that a very simple device can be used for high sensitivity mass measurements.

The authors would like to acknowledge both NANO-MASS II (EU, IST-2001-33068) and Forskningsrådet for Teknologi og Produktion (FTP-26-04-0058) for financial support of this work.

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